

Certificate of Analysis

Standard Reference Material C1252a

Phosphorus Deoxidized Copper – Cu IX

This Standard Reference Material (SRM) is intended primarily for use in evaluating chemical methods of analysis and in the calibration of instrumental methods for analysis of copper and its alloys. A unit of SRM C1252a consists of a directionally solidified, chill-cast block, approximately 32 mm square and 19 mm thick.

The certified values for 17 elements in SRM C1252a are listed in Table 1. Information values for five elements are listed in Table 2. For all elements, values are reported as mass fractions [1]. Value assignment categories are based on the definition of terms and modes used at NIST for chemical reference materials [2] and uncertainties are assessed according to the ISO/NIST Guides [3]. Table 3 summarizes the analytical methods used by NIST and cooperating laboratories for characterization of the composition of this SRM.

Certified Values and Uncertainties: A NIST-certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified value listed for a constituent is the present best estimate of the true value based on the results of analyses performed at NIST and cooperating laboratories using test methods listed in Table 3. The uncertainty listed with each value is an expanded uncertainty, with coverage factor 2, calculated by combining a between-method variance [4] with a pooled, within-method variance following the ISO and NIST Guides [3].

Information Values: The information values for the constituents of SRM C1252a are given in Table 2. These are noncertified values with no uncertainty reported because there is insufficient information with which to make the appropriate statistical assessments.

Expiration of Certification: The certification of this SRM is valid until **01 February 2027**, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). However, the certification will be nullified if the SRM is damaged, contaminated, or modified.

INSTRUCTIONS FOR USE

The test surface is the side opposite to the labeled surface. The certified portion extends from the test surface to a depth of 13 mm. Each packaged block has been prepared by finishing the test surface using a milling machine. The user must determine the optimum surface preparation procedure for each analytical technique. For example, preparation for X-ray fluorescence measurements at NIST involved fly cutting to avoid smearing of soft metals. The user is cautioned to use care when either resurfacing the block or performing additional polishing as these processes may contaminate the surface.

Coordination of the technical measurements for certification was accomplished under the direction of J.R. Sieber of the NIST Analytical Chemistry Division.

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Willie E. May, Chief Analytical Chemistry Division

Robert L. Watters, Jr., Acting Chief Measurement Services Division

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Analytical measurements for certification of this SRM were performed by T.A. Butler, M.L. Salit, J.R. Sieber, and G.C. Turk of the NIST Analytical Chemistry Division, and E.S. Beary, D.A. Becker, C. Blundell, K.A. Brletic, B.I. Diamondstone, M. Epstein, J.D. Fassett, E.L. Garner, J.W. Gramlich, R.R. Greenberg, W.R. Kelly, G.J. Lutz, L.A. Machlan, J.R. Moody, P.J. Paulsen, and T.C. Rains of the NBS Inorganic Analytical Research Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.M. Adams of the NIST Measurement Services Division.

Stability: This material is considered to be stable during the period of certification. NIST will monitor this material and will report any significant changes in certification to the purchaser. Registration (see attached sheet) will facilitate notification.

Table 1. Certified Values for SRM C1252a Phosphorus Deoxidized Copper – Cu IX

Constituent	Mass Fraction (mg/kg)			Constituent	Mass Fraction (mg/kg)*			
Sb	42	±	1	Ni	128	±	1	
As	118	\pm	2	P	125	\pm	21	
Cd	16.9	\pm	1.3	Se	56	\pm	8	
Co	87	\pm	4	Aq	158	\pm	10	
Cv	19	\pm	2	Te	54.6	\pm	2.5	
Au	33.9	\pm	1.4	Sn	120	\pm	12	
Fe	72	\pm	9	Zn	69.4	\pm	1.0	
Pb	60	\pm	1	Cu	99.87 %	<u> </u>	0.13 %	
Mn	43	\pm	7					

^{*}Note copper is given in units of mass fraction (%), not mg/kg.

Table 2. Information Values for SRM C1252a, Phosphorus Deoxidized Copper – Cu IX

Constituent	Mass Fraction (mg/kg)	Constituent	Mass Fraction, (mg/kg)		
Al	< 20	Si	<100		
Bi	19	S	70		
Mg	< 20				

Material Preparation: SRM C1252a Phosphorus Deoxidized Copper is one in a series of twelve different copper composition "Benchmark" materials. The series consists of Cu "O" through Cu "XI" that was prepared in a cooperative NBS-ASTM-Industry Program. The base material for the preparation of Cu IX was vacuum melted and cast into a single ingot at Canon Muskegon Corp., Muskegon, MI. About 25 elements were included in the aim composition, covering the concentration range of about 15 mg/kg to 500 mg/kg. The final base material for SRM C1252a, Cu IX, was prepared by remelting and recasting portions of the original ingot sections on the NIST water-cooled, copper plate mold assembly at the Brass Foundry, American Cast Iron Pipe Co., Birmingham, AL. The preparation and homogeneity testing plan was similar to that described in NBS Miscellaneous Publication [5]. Extensive homogeneity studies were performed at NIST Boulder, by J.G. Hust using residual resistivity ratio measurements and at NIST Gaithersburg, by C.H. Brady using metallographic studies, and by G.J. Lutz using neutron activation analysis. The results of measurements indicated the maximum material variability to be less than 10 %.

Cooperating Laboratories: The following laboratories performed cooperative analyses for material characterization and certification during the period from 1975 to 1985:

Anaconda Company, Primary Metals Division, Raritan Copper Works; Perth Amboy, NJ

Kennecott Copper Corporation, Metals Mining Division; Salt Lake City, UT and Utah Copper Division; Magna, UT Kennecott Refining Corporation; Baltimore, MD

Phelps Dodge Refining Corporation, El Paso Works; El Paso, TX

Reading Metals Refining Company; Carteret, NJ

U.S. Metals Refining Company, AMAX Copper Division; Carteret, NJ

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Table 3. Methods of Analysis for SRM C1251a, SRM C1252a, and SRM C1253a

Element	Methods*									
Element	A	В	С	D	Е	F	G	Н	I	J
Al	X				X			X		
Sb						X		X		
As			X			X		X		
Bi			X					X		
Cd	X				X			X		
Co						X		X		
Cu								X	X	X
Cr						X		X		
Au		X						X		
Fe					X	X		X		
Pb				X				X		
Mg								X		
Mn					X			X		
Ni			X	X				X		
P					X		X	X		
Se				X		X		X		
Si	X						X	X		
Ag		X				X		X		
S								X		
Te				X				X		
Sn	X		X					X		
Zn						X		X		

*Methods:

- A. Atomic Absorption Spectrophotometry
- B. Fire Assay
- C. Photon Activation Analysis
- D. Isotope Dilution Mass Spectrometry
- E. Direct-Current Plasma Atomic Emission Spectrometry
- F. Neutron Activation Analysis

- G. Chemical Analysis
- H. X-ray Fluorescence Spectrometry
- I. Inductively Coupled Plasma Atomic Emission Spectrometry
- J. Electrogravimetry

REFERENCES

- [1] Taylor, B.N.; Guide for the Use of the International System of Units (SI); NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (1995).
- [2] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC, p. 16 (2000).
- [3] ISO; Guide to the Expression of Uncertainty in Measurement; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.
- [4] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*, J. Res. Natlo. Inst. Stand. Technol., Vol. 105, No. 4, p. 571, (2000).
- [5] Michaelis, R.E.; Wyman, L.L.; Flitsch, R.; Standard Reference Materials: Preparation of NBS Copper-Based Spectrochemical Standards, NBS Misc. Publ. 260-2, U.S. Government Printing Office: Washington, DC (1964).

Certificate Revision History: 17 June 2004 (This editorial revision adds a % sign to Table 1); 23 July 2002 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

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